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by

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ABSTRACT

Pieces of asbestos-cement pipes obtained from water distribution systems and laboratory investigations were studied with a scanning electron microscope, an X-ray analysis unit, and two types of hardness testing instruments. Rockwell "L" hardness measurements and modified Shore "D" durometer readings were found to be related to the depth to which calcium had been lost from a pipe exposed to aggressive water. The calcium loss was measured by an X-ray analysis technique performed in a scanning electron microscope. The modified durometer showed promise as a field instrument for determining the extent to which asbestos-cement pipe had been attacked by aggressive water.

INTRODUCTION

Asbestos-cement (A/C) pipe has been widely used in drinking water distribution systems throughout the world for over 50 years. Although the problems associated with the corrosion of metal piping materials in potable water systems have been the subject of numerous studies, asbestos-cement materials generally had been considered unaffected by potable waters.

When the association between occupational exposure to asbestos and health problems was determined (1), questions were raised about the release of fibers from A/C pipe (2). After a series of field tests, tank tests, and a pipeloop experiment, Buelow et al. (3) concluded that although A/C pipe had had a reputation for resisting attack by aggressive waters that cause corrosion of metal pipe materials, it actually behaved much like other piping

materials, except plastic pipe, that are commonly used for the distribution of drinking water. They concluded that A/C pipe would corrode and deteriorate if exposed to aggressive water. Presently, a clear understanding of all the factors involved in causing a water to be aggressive or non-aggressive to A/C pipe is not available. This is partly due to the lack of quantitative methods of assessing the condition of A/C pipe. Pipes exposed to different water qualities or treatment practices can only be roughly compared with the presently used methods.

In this study, four analytical techniques were evaluated as to their usefulness in assessing the condition of A/C pipe in a quantitative manner. Two of the techniques involved standard materials hardness tests: the Rockwell "L" Scale and the Shore "D" durometer. The other two techniques utilized a scanning electron microscope (SEM).

BACKGROUND

Judgments on the deterioration of asbestos-cement pipe must be made on two levels: loss of structural integrity and potential for release of asbestos fiber into the drinking water. Reduction of structural integrity to the pipe occurs only after corrosion has taken its toll on a substantial part of the bulk pipe material. Release of asbestos fiber into the water, however, may occur after corrosion has affected only a few hundred micrometers of the interior surface of the pipe.

Standard methods exist for testing the structural integrity of new A/C pipe (4) and have been used in studies of how resistant the pipe is to attacks by sulfate environments (5). The American Water Works Association with the help of the asbestos-cement pipe producing industry published an Aggressive Index (A.I.) (Figure 1) based on calcium carbonate saturation considerations to help utilities predict when water should be considered harmful to the structural integrity of A/C pipe (4). No standard analytical methods or water quality guidelines presently exist for evaluating the potential for an asbestos-cement pipe to release fiber into the water. Because of the desire to predict the occurrence of fibers in drinking water, there has been a tendency to use the A.I. as an indicator of fiber release from the A/C pipe. Some initial data published by McFarren et al. (6) provided evidence of a correlation between fibers in the drinking water of A/C pipe distribution systems and the A.I.. They reported finding asbestos fiber in two systems with aggressive indices below 10 and no fibers in three systems with aggressive indices above 12.

The limitations of the A.I. in predicting calcium carbonate saturation and precipitation over the entire pH range have been described by Rossum and Merrill (7). Similar theoretical considerations led Schock and Buelow (8) to criticize the A.I. as an indicator of corrosivity and fiber release from A/C pipe. They concluded that:

> Insofar as the A/C pipe would indeed be protected by a film of $CaCO_3$ if active $CaCO_3$ precipitation were occurring, the A.I. would then give a fairly accurate prediction of nonaggressiveness. However, in the case of calcium carbonate undersaturation, the aggressiveness of the water toward the pipe will be dictated

by other chemical equilibria. Calcium carbonate is, at a maximum, a trivial component of the matrix of A/C pipe (Troxell et al., (9)). Therefore, there is no a priori reason to expect the A.I. as traditionally conceived to give an accurate prediction of the pipe's interior surface condition in waters undersaturated with respect to calcite.

When attempts are made to predict the amount of pipe softening or calcium leaching from the pipe interior by using the A.I. of the water, additional error is introduced by implying a correlation between the magnitude of calcite undersaturation and the rate of dissolution of the predominantly calcium-silicate pipe. A correlaation between faster calcium leaching and greater calcite undersaturation is, more probably, a coincidence resulting from some kinetic effect of calcium concentrations in the waters (and probably the pH) with regard to calcium-silicate or calcium-hydroxide saturation.

They also noted that the A.I. might falsely indicate fiber release when the pipe was actually being protected by chemical reactions aside from calcite deposition. Research has shown that zinc (10) and iron (11) can provide protection in undersaturated conditions.

The A.I. is, however, attractive to water supply personnel because it is simple and inexpensive to determine. At present, it is the only general water chemistry model being used for predicting the behavior of asbestos-cement pipe in water distribution systems. The development of a more accurate model and the evaluation of corrosion inhibition treatments require good methods for quantitative comparisons between asbestos-cement pipes exposed to waters of different qualities. In addition to research analytical methods, utility personnel are also interested in methods for use in the field for assessing the potential for asbestos fiber release from their water systems.

Several investigators have looked for asbestos fibers released to the water flowing through A/C pipe as an indication of corrosion of the pipe (6,12-16). This is tedious and subject to interference from fibers contributed to water by other sources.

Schock and Buelow (8) and Kinman (17) both used visual examination of the pipe surface to draw conclusions about the corrosiveness of waters on A/C pipe coupons they were testing. Visual examination is, of course, a subjective observation and not easily quantifiable for careful research comparisons of treatments.

Buelow et al. (3) described a fingernail scratch test when evaluating the softening of the A/C pipe interior. The procedure consisted simply of determining whether a pipe surface could be scratched with a fingernail or not. Fingernail scratch test can be used in the tentative identification of minerals in the field (18). A fingernail has a hardness of about 2.5 on the Mohs' Scale, a scale of relative hardness based on the ability of one substance to scratch another. Gypsum has a value of 2 on that scale and calcite, a value of 3. New A/C pipe cannot be scratched with a fingernail but a severely attacked piece of pipe can be scratched easily. Although this measure is

useful in the field, it is difficult to use it to quantify the degree of attack for comparison with another piece of A/C pipe.

In order to find a more reproducible, less subjective test of the physical condition of A/C pipe, the City of Bellevue, Washington, under an EPA grant, delivered several A/C pipe sections to a testing laboratory (Northwest Laboratories of Seattle, Inc.). A number of test procedures, including Brinnell hardness tests and various scales of the Rockwell hardness testing procedure were evaluated. The Shore "D" durometer test and Rockwell hardness test utilizing the "L" scale showed differences between used and new pipe (19).

Evaluation of Testing Procedures

Four testing procedures, two hardness tests as described in the literature and two techniques developed for the SEM, were evaluated by applying them to samples of asbestos-cement pipe from both the field and from experimental test systems. Tables 1 and 2 list some characteristics of the pipe pieces used. A more detailed description for most of the field systems will be provided elsewhere (20).

Rockwell Hardness Testing

In Rockwell testing a hardness number is obtained representing the additional depth of penetration into a material to which a steel ball is driven by a heavy load, beyond the depth of a previously applied light load. The minor load is applied first. Then the major load is applied. While the piece being tested is held in a fixed position, the major load is removed and the hardness number automatically indicated on the dial guage (Figure 2).

Because there are no standard Rockwell scales for asbestos-cement materials, an "L" scale used for plastics and hard rubbers was used (21). The Rockwell "L" test uses a 1/4 inch ball penetrator and a 60 kilogram-force load. The B dial scale generally used with soft material was used with the A/C pipe. Corroded A/C pipe has a tendency to act like some hard rubbers and change slightly under sustained uniform pressure. As suggested in the Rockwell manual, a time factor was established; the load application was exerted for a duration of 10 seconds before the value was read. The Rockwell testing procedure was repeated at least 5 times on each sample.

The effect of the moisture content of A/C pipe materials on the hardness test results has been studied by Ramaley (22). From a series of tests, he concluded that the hardness values increased in the range of 5 to 10 percent with drying to constant weight. He also found that the hardness test results were very similar for both air dried and oven dried coupons.

The Rockwell test was found to be sensitive to how the sample was placed on the anvil. According to the discussion in the ASTM Standard Test Method for Rockwell Hardness (21), tubular or unsupported curved plastic specimens are not recommended for hardness testing. The standard test method does not consider asbestos-cement material but the curved A/C pipe samples were also found to give an erroneous result unless the hardness value was determined along a line at the center of the pipe piece. From one six-inch diameter pipe

coupon the center line hardness values ranged from 64 to 73 (all values were within 6% of the mean) but the values decreased from 64 to 28 for measurements taken on a line across the sample, perpendicular to the center line.

The results of replicate Rockwell hardness values on the samples were all within 10% of the mean value and most were within 5%. Some field pipe samples showed evidence of softening on the outside of the pipe. To learn if this might have affected the Rockwell hardness values tests were performed with a thin foam rubber mat placed under a piece of new A/C pipe. This simulated softened back surface did not change the hardness values more than the 10% variability found in the measurement.

Durometer Hardness Testing

The durometer hardness test is based on the penetration of a specified indentor forced into a material as registered on a dial by means of a calibrated spring. The indentation hardness is inversely related to the penetration and is dependent on the elastic modulus and viscoelastic behavior of the material. The method is considered an empirical test method intended primarily for control purposes (23). The potential importance of the durometer in testing asbestos-cement pipe is that it can be used as a hand held instrument and therefore could be used in the field.

The first durometer tested was a Model 307L Type "D" Scale durometer as suggested in Ramaley (22).† However, the surface of the pipe specimens were not flat as required by the AST Standard D2240 (23) for durometer test specimens. As can be seen in Figure 3 the flat bottom plate of the standard durometer model did not fit the pipe specimens. In order to overcome the curvature problem, Pacific Transducer was engaged to specially modify a durometer base so that it would fit the curvature of a four-inch or larger diameter pipe. The modified durometer was of a size that it would fit into a six-inch diameter or larger asbestos-cement pipe without requiring that the pipe be cut. A comparison of the bases of the two durometers can be seen in Figure 4.

The modified durometer was not affected by a foam rubber mat backing on a pipe piece. The results for replicate durometer readings on the same sample were generally within 7% of the mean value. On two occasions large sand crystals were found to interfere with the measurement and give a reading 15% higher than the mean value. In an investigation of the variability of the hardness around the inside of a pipe, four whole pipe pieces from the field samples were tested at 10 points around their inside diameter. The maximum variability of hardness was 5% from the mean value. The orientation of the durometer was found to be important to accurate readings. If the long axis of the rounded base of the durometer was not parallel to the long axis of the pipe curvature, inaccurate low values were obtained. However, as long as the long axis of the instrument is parallel to the pipe, readings may be obtained anywhere on the pipe interior.

Hardness values for the four coupons from the Bellevue experimental tank tests had been determined independently by Ramaley (22). A comparison with

†Pacific Transducer Company, Los Angeles, California

the values determined in this study are shown in Table 3. The differences between the durometer readings are apparently caused by the differences in the durometer bases. Figure 4 shows the problem of using a flat based durometer with a curved pipe section. The values for the Rockwell hardness measurements show little difference between the two labs.

Scanning Electron Microscopy (SEM)

A Perkin-Elmer ETEC Autoscan Scanning Electron Microscope was used to study the A/C pipe samples (Figure 5). A special holder was machined to hold a pipe sample as large as six inches long and two inches wide so that the coupons could be analyzed with as little cutting as possible. The holder was constructed with a position indicator to aid in placing the coupons in the SEM reproducibly when they were analyzed more than once.

While SEM electron micrographs can show dramatic differences between the surfaces of new and seriously deteriorated A/C pipe (24, 25), they do not provide a comparison which is quantitative. A measure of the number of fibers exposed on the surface of the pipe is a more useful comparison.

A method for measuring fiber cover was adapted from a method used in petrology for estimating mineral volume percentages (25). The edges of a randomly chosen photographed area are traced onto transparent plastic sheets using a marker. The area covered by fibers or fiber bundles is then outlined. When all the fibers are accounted for, the photograph is removed and a sheet of squared graph paper is placed under the overlay. The number of squares more than half covered by the fiber area is divided by the total number of squares to give the fraction of area covered by exposed fibers. In new A/C pipe only a few fibers are visible on the pipe surface (Figure 6), but as the cement binder is dissolved or eroded away more fibers are visible. In cases of severely attacked pipe as shown in Figure 7, the area not covered with fibers was measured and substracted from the total area. There are no standard specimens that would be useful for checking this technique. As shown in Table 4, new asbestos cement pipe had consistently low numbers of fibers in the areas analyzed. A pipe coupon that had been exposed to very aggressive water had a variability in exposed fibers of about 10%. A series of pieces (1, 2, and 3) taken from a 3 foot length of distribution pipe which visually appeared to have both corroded and smooth areas showed considerably more variation.

The estimate of exposed fibers on the surface of the pipe was time consuming and the variability was considerable for low values. For a mean value of 3% the range was between 0 and 4%. For the exposed fibers above 10%, the upper and lower values were generally within 50% of the mean. This high variation is a result of the non-homogeneity of the asbestos-cement material. The percentages given for exposed fibers are useful as indicators but the variation in the values must be considered when comparing them. Local corrosion or coating also caused great variation in exposed fiber values. As shown in Table 4, an area of a distribution pipe (Distribution 3) which showed evidence of being coated had only 4% of the surface showing exposed fibers but another sections (Distribution 1 and 2) where there was evidence of the coating tearing away showed 31% and 44% fibers exposed. The effect of

operator bias in this technique has not been tested and may be an important source of variability.

The second SEM technique followed from some earlier work by one of us (Logsdon) using an optical microscope to examine cross-sections of A/C pipe mounted in bakelite (26). He had reported seeing differences in the color and texture of the inner wall layers of a pipe exposed to aggressive water in a distribution system from Wisconsin.

Experiments showed that the mounted cross-section of pipe (Figure 8) could also be used to study changes in elemental composition from the bulk material of the pipe to the inner surface of the pipe. This was done by elemental line scanning using the X-ray energy dispersive analysis in the SEM. In this analysis, after an elemental spectrum is acquired, the element of interest (calcium, iron, silicon) is defined by a region of interest on the spectrum display.

An element line scan was then done on the piece. The trace time (500 seconds) was co-ordinated with the SEM scan time. A photograph of the line scan (Figures 9 and 10) was compared to a photograph of the pipe cross-sectional area analyzed and the linear relationship between the scan distance and the line scan was calculated. Measurement of the thickness of a coating or depth of loss of an element was then determined. The quantitative analysis depends on a measurement from a photograph of an X-ray scan. The calcium line scan and the silicon line scan are obtained without any movement of the pipe sample. If the calcium trace fell below the silicon trace, it was an indication of calicum lost. A calcium trace extending further than the silicon trace suggested a calcium coating. The measurement was made in each case from from a photograph of the line scans of calcium and silicon displayed simultaneously. Several measurements were made from each piece of pipe and an average value determined. The cross-sections of several pieces of new pipe were analyzed to check the system. There was no decrease in calcium with any of the new pipe analyzed (Figure 9).

The only available data on the variation of the calcium content of the pipe material as a function of the distance from the inner surface of the pipe was that published by Kristiansen (27). He had cut samples of the inner portion of the material from a cross-section of his experimental pipe into sections 0.5 mm thick and analyzed these sections separately. The percent of calcium as CaO determined for each section is given in Table 5. Dr. Kristiansen very kindly sent a piece of his pipe for analysis. An elemental scan was performed across the cross-section as described previously. In order to compare the calcium concentrations determined by the two methods, the data by Kristiansen was converted to a percentage of calcium of the bulk material and then plotted along with every fifth point of the X-ray scan as shown in Figure 11. With the exception of the inner most section, the data agree extremely well. Additional scans varied very little from the first scan.

RESULTS AND DISCUSSION

A relationship was found between the Rockwell and modified durometer hardness tests and the depth to which calcium was leached from the pipe

(Figures 12 and 13). Linear regression analysis produced a correlation coefficient of 0.953 for the relationship between the modified Shore "D" durometer and calcium loss as measured by the X-ray scan and a correlation coefficient of 0.928 for the Rockwell hardness and calcium loss data.

Neither parameter, hardness or depth of calcium loss, was related to the measure of fiber exposure in a direct linear way. The linear correlation coefficients were all less than 0.40. This was not surprising because the evidence of attack on the pipe (calcium loss or pipe softening) does not consider coatings that have independent effects on the number of exposed fibers on the pipe surface. As might be expected, it was observed that both soft pipe with evidence of iron coating and hard pipe with no coatings had few fibers on the surface. However, the field and laboratory pipes samples examined in this study did not provide enough data to adequately study the effect of the interactions between the two factors on the amount of exposed fibers on the pipe surface.

The modified durometer showed promise of being a useful tool for assessing the condition of A/C pipe in the field. The durometer hardness values were found to be related to the loss of calcium from the pipe as determined by X-ray elemental profile scans of pipe cross-sections. The depth of calcium loss determinations made with the X-ray technique performed in a scanning electron microscope compared favorably with the determinations made using bulk chemistry methods in another laboratory. The modified durometer reading, therefore, is a numerical indication of the extent to which calcium has been leached from a pipe wall by corrosive waters. The reading is not, however, directly related to the amount of exposed fibers on the surface of the pipe because the pipe may be coated with iron or another material which binds the fibers to the surface but does not completely prevent calcium leaching. For an iron coated pipe, a low modified durometer hardness value would indicate a softened pipe which may be susceptible to local corrosion problems such as tearing of the coating and exposure of fibers in areas of the pipe exposed to flowing water with changing turbulence patterns.

CONCLUSIONS

The hardness tests, Rockwell and modified durometer, and the X-ray scan measurement of calcium loss were found to give quantifiable measures of the degree to which asbestos-cement pipe had been attacked by aggressive water but these values were not related to the amount of exposed fiber on the surface of the pipe and therefore could not be used to predict the potential for fiber release from the pipe.

The modified durometer could be used in the field to compare in service A/C pipe with new pipe to determine whether or not it had been attacked by aggressive water.

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TABLES

TABLE 1. A/C Pipe Samples from the Field
TABLE 2. A/C Pipe Samples from Experimental Studies
TABLE 3. Comparison of Hardness Test Results
TABLE 4. Percent of Area With Exposed Fibers
TABLE 5. Pipe Calcium Data Determined by Kristiansen (27)

Table 1.
A/C Pipe Samples from the Field

Water Distribution System	Aggressive Index	Visual Evidence of Coat	Age of Pipe
J*	5.7	no	5 yrs
K	7.5	yes	15 yrs
L	9.5	yes	10 yrs
M	10.0	no	40 yrs
N	10.4	yes	20 yrs
I*	11.6	yes	
O	11.8	partial	15 yrs
P	12	no	10 yrs
O	>12	yes	18 yrs
R	13	yes	20 yrs

*same systems I and J as in Buelow et al. (1)

Table 2.
A/C Pipe Samples from Experimental Studies

Investigator	Pipe Code	Description	Initial A.I.	Iron in Water mg/L	Velocity ft/sec
1. R. Kinman (17)		Single Pass System			
A-2-O	AO		13.1		0.27
C-1-F	CF		12.1		0.1
2. R. Buelow (1)		Recirculating			
Pipeloop	PI		7.2	Much	1.7
Exp. 1	E1		10.2	<0.1	1.0
Exp. 3	E3		9.3	<0.1	1.0
Exp. 19	E19		9.4	0.1	1.0
3. H. Kristiansen (27)		Single Pass System			
7 yrs Oslo Pipe	OS		8.1		Low
4. B. Ramaley* (22)		Recirculating			
T1-2	T1		9.5	<0.1	1.0
T3-2	T3		10.4	0.1	1.0
T5-2	T5		10.5	<0.1	1.0
T8-2	T8		10.0	0.3	1.0

*Investigator for J. M. Montgomery Engineers on an EPA sponsored project in Bellevue, WA.

Table 3.
Comparison of Hardness Test Results

Sample Coupon	Shore 'D' Hardness		Rockwell 'L' Hardness	
	Present Study*	Ramaley (22)**	Present Study	Ramaley (22)
T1-2	93	88	94	97
T3-2	92	82	94	93
T5-2	94	86	97	95
T8-2	91	82	94	92

*Modified durometer
**Standard durometer

Table 4.
Percent of Area With Exposed Fibers

Areas	1	2	3	4	5	Mean
New Pipe 1	2%	<1%	1%	1%	2%	1%
New Pipe 2	<1%	2%	2%	<1%	1%	1%
Test Coupon	27%	35%	43%	40%	32%	35%
Distribution 1	24%	28%	42%	33%	29%	31%
Distribution 2	34%	54%	48%	39%	46%	44%
Distribution 3	5%	<1%	7%	5%	2%	4%

Table 5.
Pipe Calcium Data Determined by Kristiansen (27)

Pipe Layer	Calcium as CaO % of Total	Calcium as Ca % of Ca in Bulk
Inside 0.5 mm	12.0	25.8
2nd 0.5 mm	12.2	26.2
3rd 0.5 mm	21.9	47.1
4th 0.5 mm	35.6	76.6
Remaining	46.5	100.0

FIGURES

FIGURE 1. Aggressiveness Index (A.I.)
FIGURE 2. Rockwell Hardness Tester (a. front view. b. side view, arrow pointing to the 60 kg mass)
FIGURE 3. Durometer Hardness Tester
FIGURE 4. Comparison of Durometers. (a. modified base (left), standard base (right). b. modified durometer. c. standard durometer.)
FIGURE 5. Scanning Electron Microscope and X-Ray Analysis Unit
FIGURE 6. SEM Measure of Fibers Exposed on the Surface of New Pipe
FIGURE 7. SEM Measure of Fibers Exposed on the Surface of Corroded Pipe
FIGURE 8. Bakelite mount of asbestos-cement pipe cross section. The arrow shows the direction of X-ray line scan from the bulk pipe to the inner pipe surface.
FIGURE 9. X-Ray Elemental Profile: New Pipe
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FIGURE 12. Calcium Loss Vs. Modified Durometer Reading
FIGURE 13. Calcium Loss Vs. Rockwell "L" Scale

$$A.I. = pH + \log_{10} (A \times H)$$

Where: pH is measured in recognized pH units
A is total alkalinity in mg/L as $CaCO_3$
H is calcium hardness, Ca in mg/L as $CaCO_3$

Interpretation: A.I. < 10 very aggressive
10 < A.I. < 12 moderately aggressive
A.I. > 12 nonaggressive

FIGURE 1. Aggressiveness Index (A.I.)

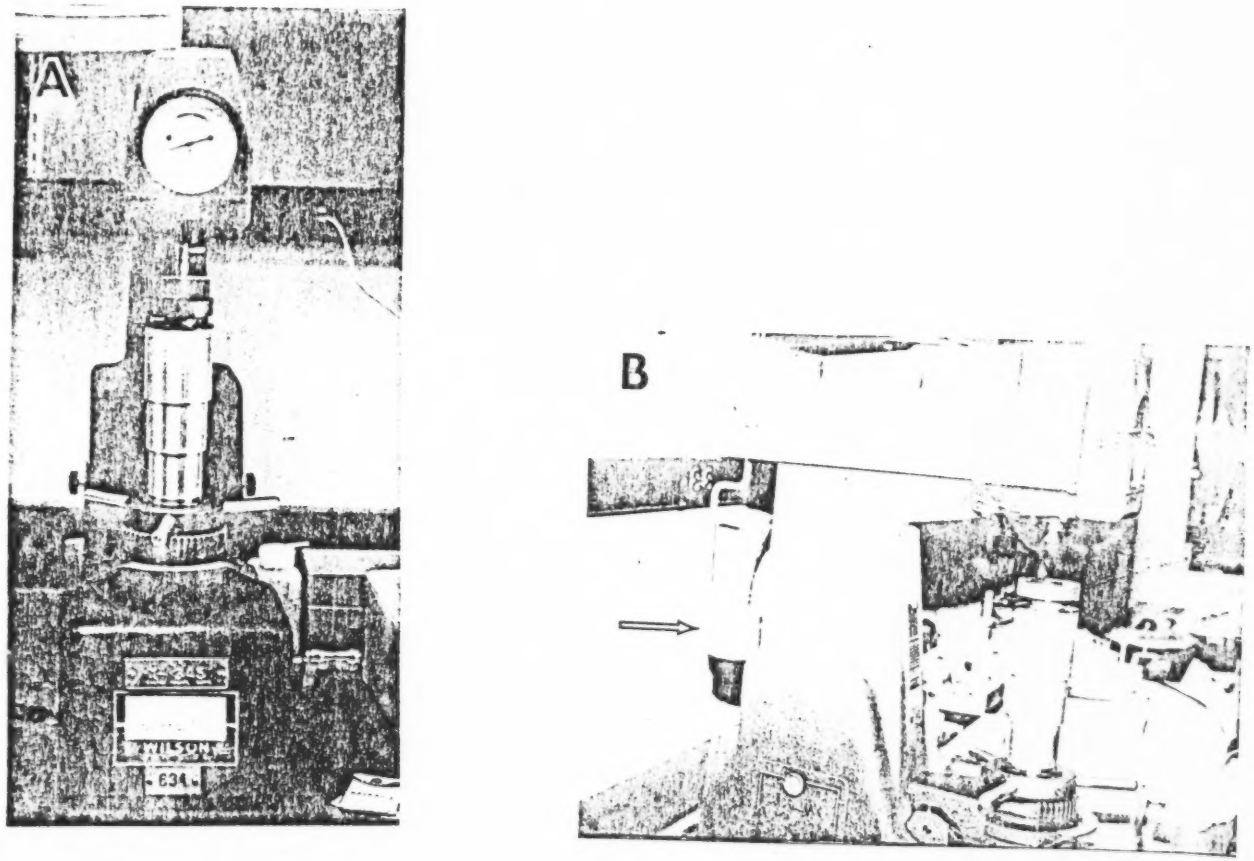


FIGURE 2. Rockwell Hardness Tester (a. front view. b. side view, arrow pointing to the 60 kg mass)

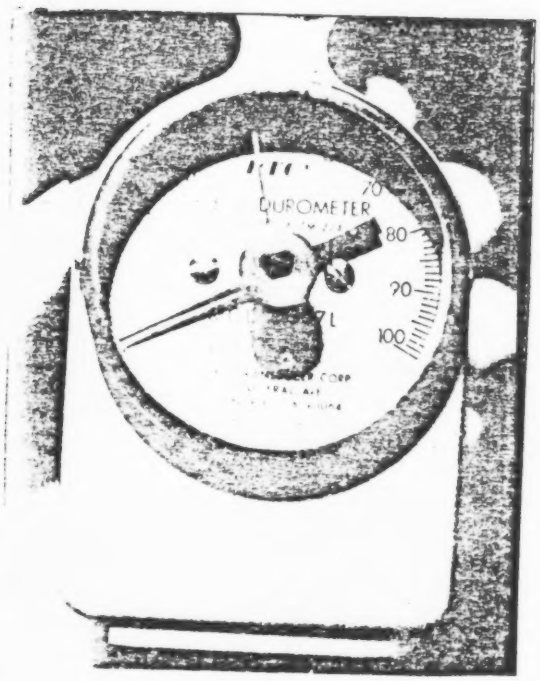
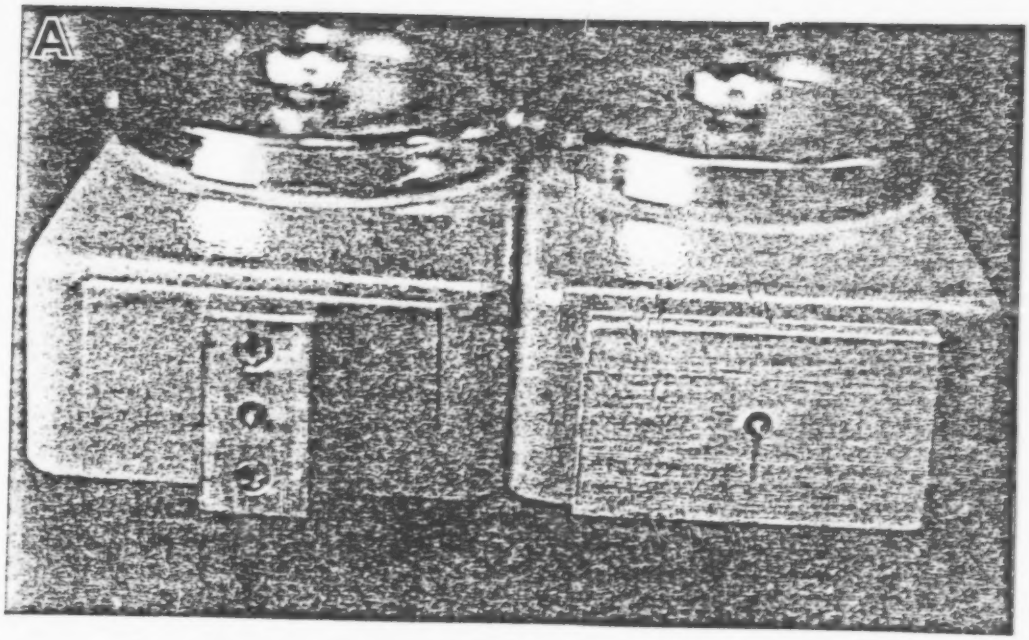
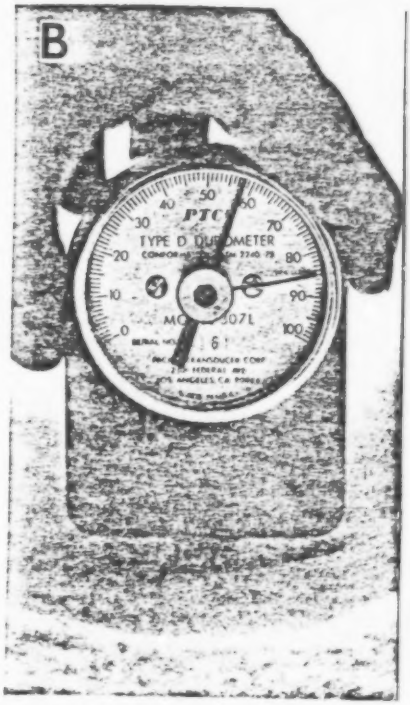


FIGURE 3. Durometer Hardness Tester





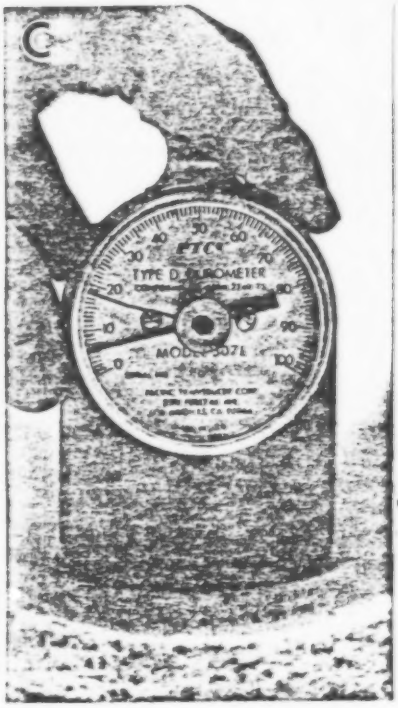


FIGURE 4. Comparison of Durometers. (a. modified base (left), standard base (right). b. modified durometer. c. standard durometer.)

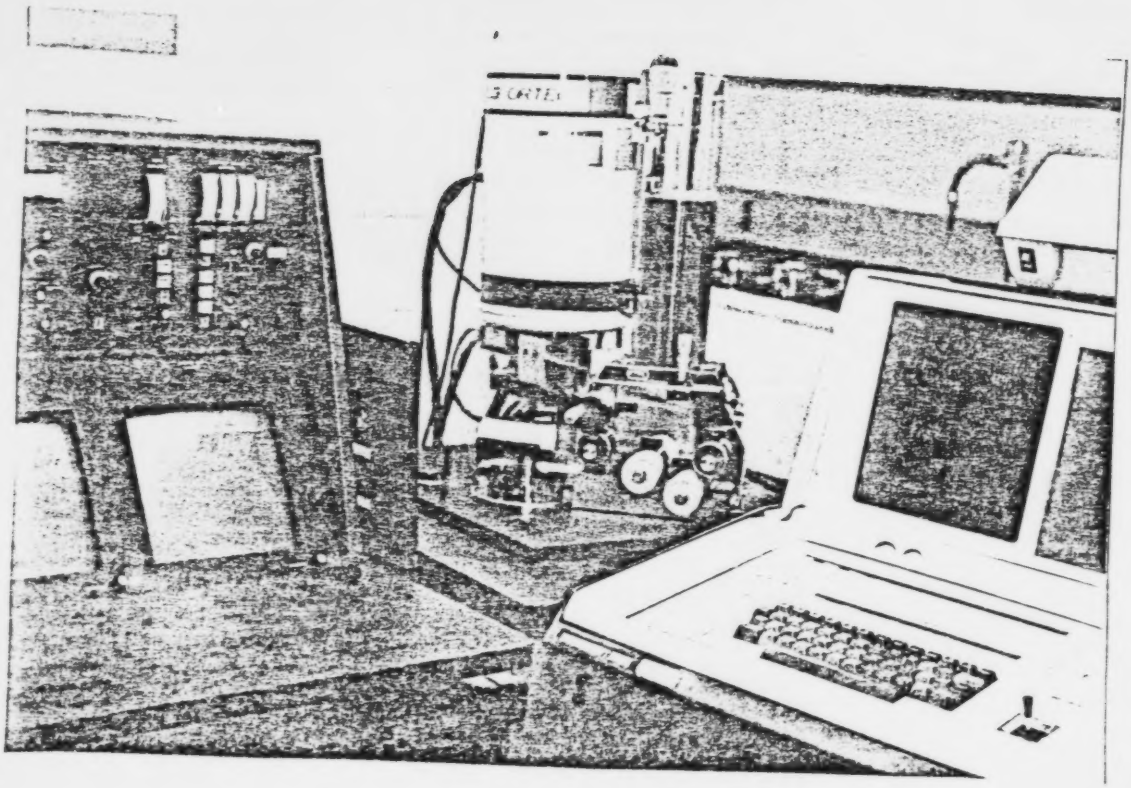


FIGURE 5. Scanning Electron Microscope and X-Ray Analysis Unit

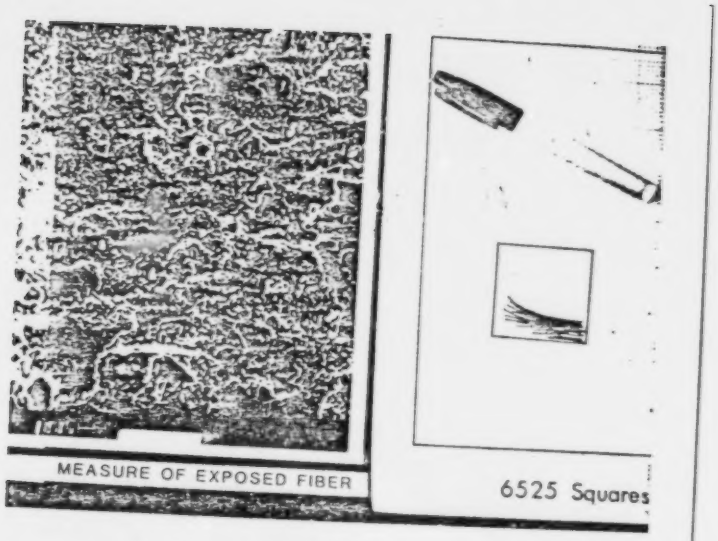


FIGURE 6. SEM Measure of Fibers Exposed on the Surface of New Pipe

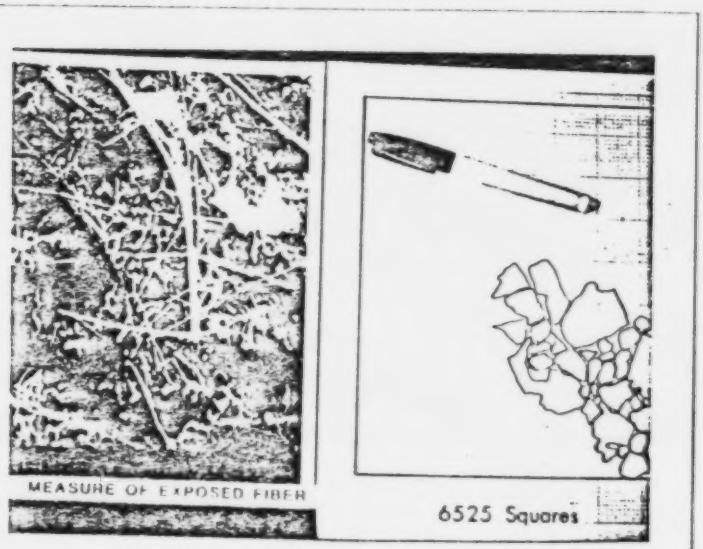


FIGURE 7. SEM Measure of Fibers Exposed on the Surface of Corroded Pipe

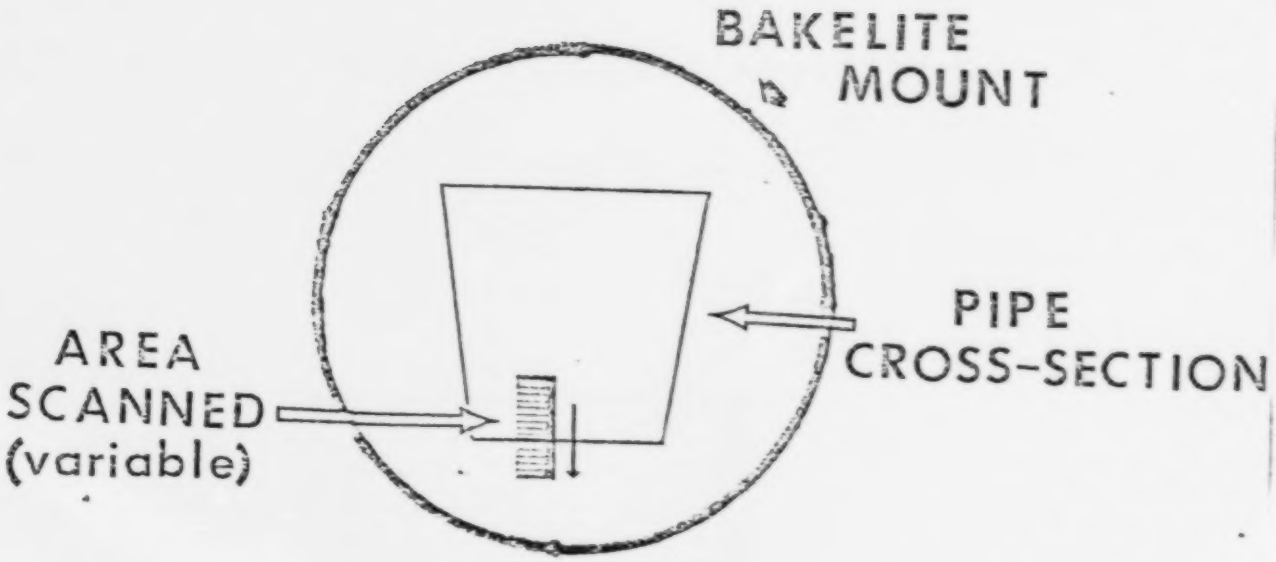


FIGURE 8. Bakelite mount of asbestos-cement pipe cross section. The arrow shows the direction of X-ray line scan from the bulk pipe to the inner pipe surface.

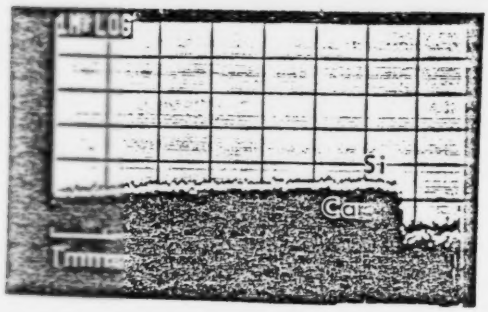


FIGURE 9. X-ray Elemental Profile : New Pipe.

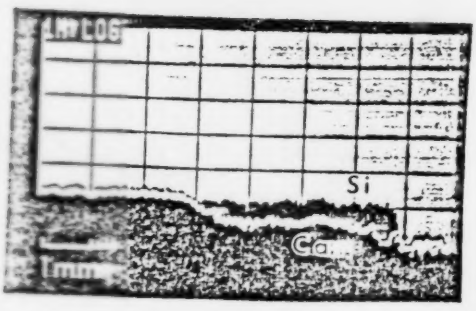


FIGURE 10. X-ray Elemental Profile : Attacked Pipe.

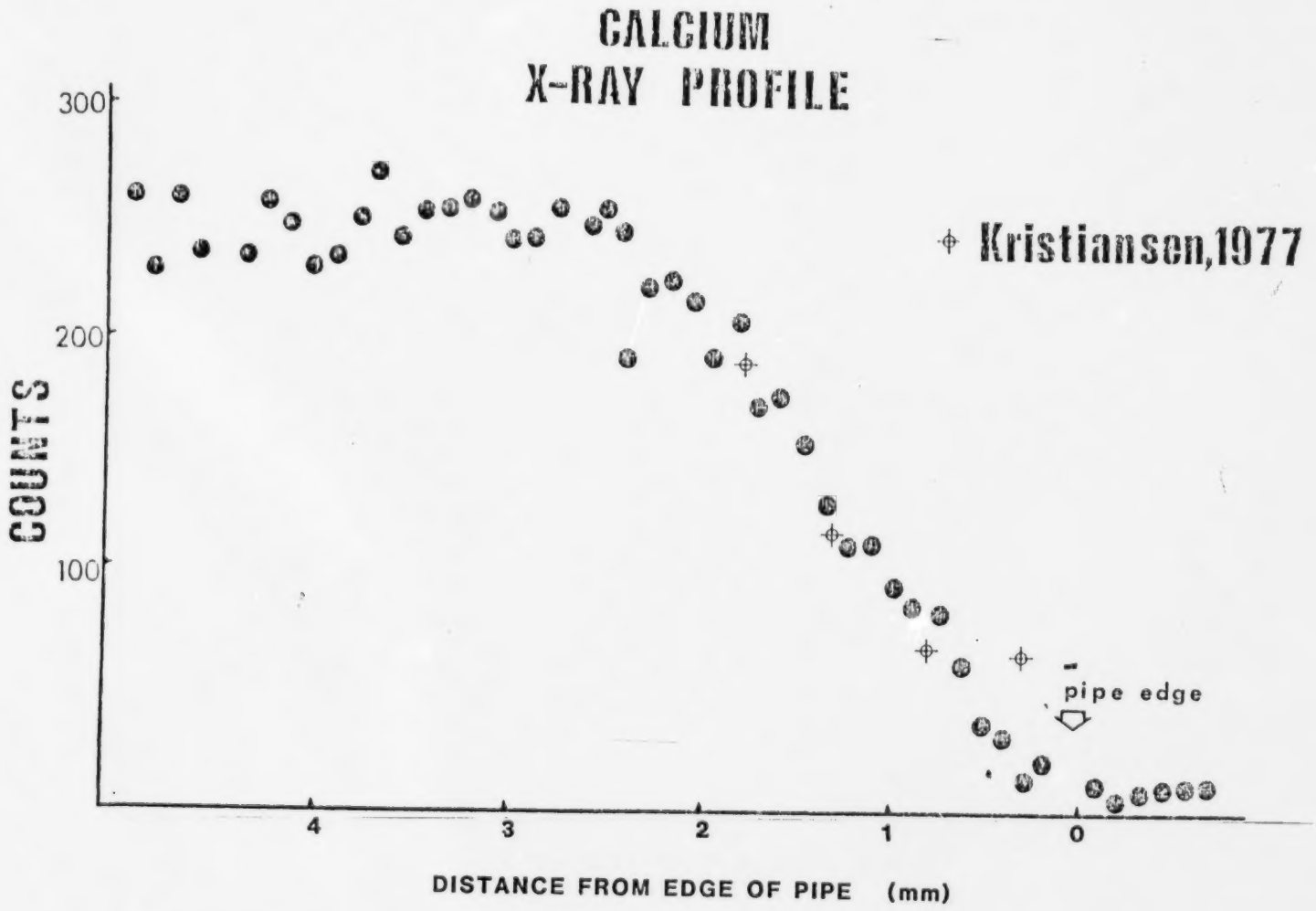


FIGURE 11. Comparison of X-Ray Calcium Profile with Kristiansen Bulk Chemistry Data

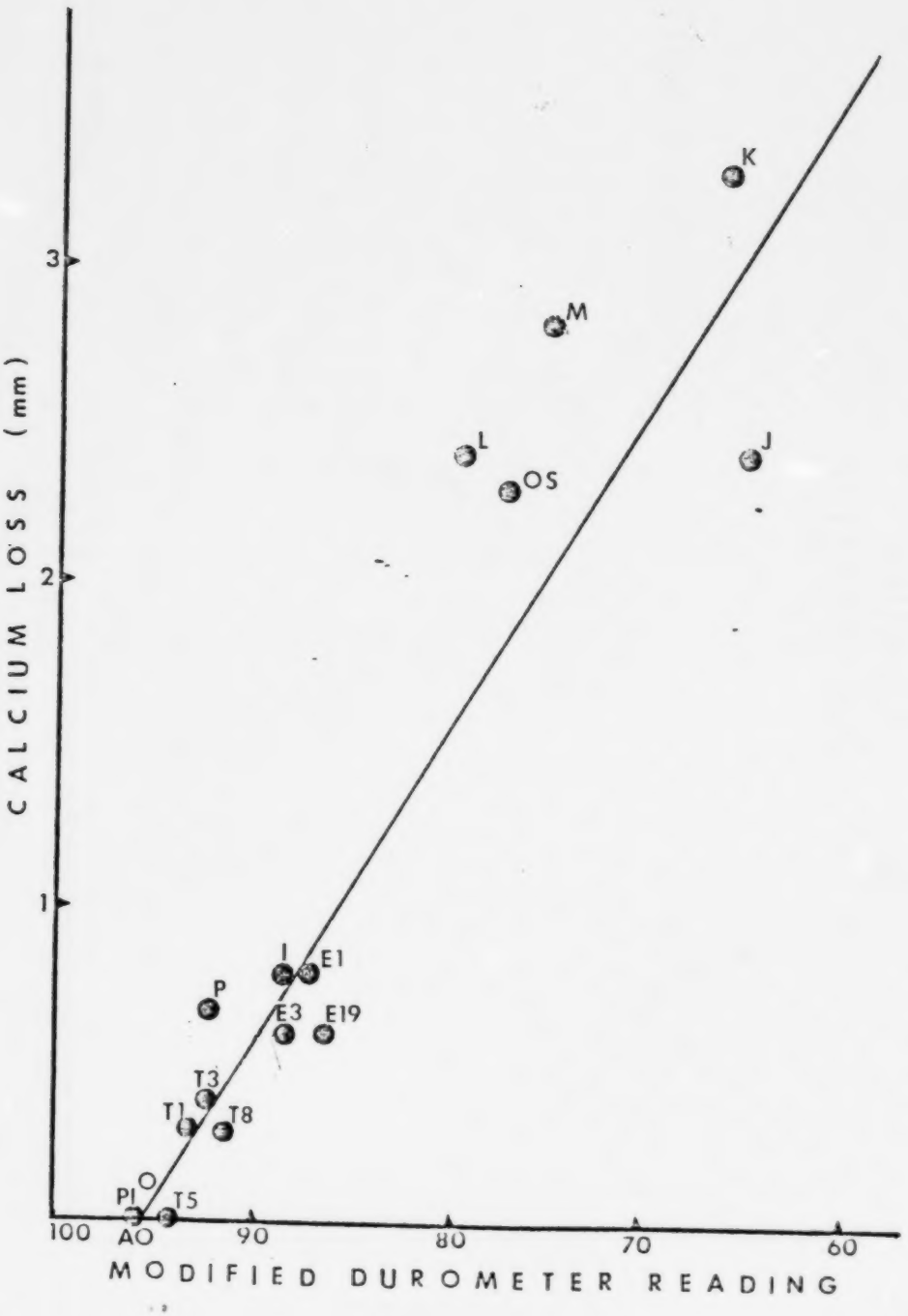


FIGURE 11. Calcium Loss Vs. Modified Durometer Reading

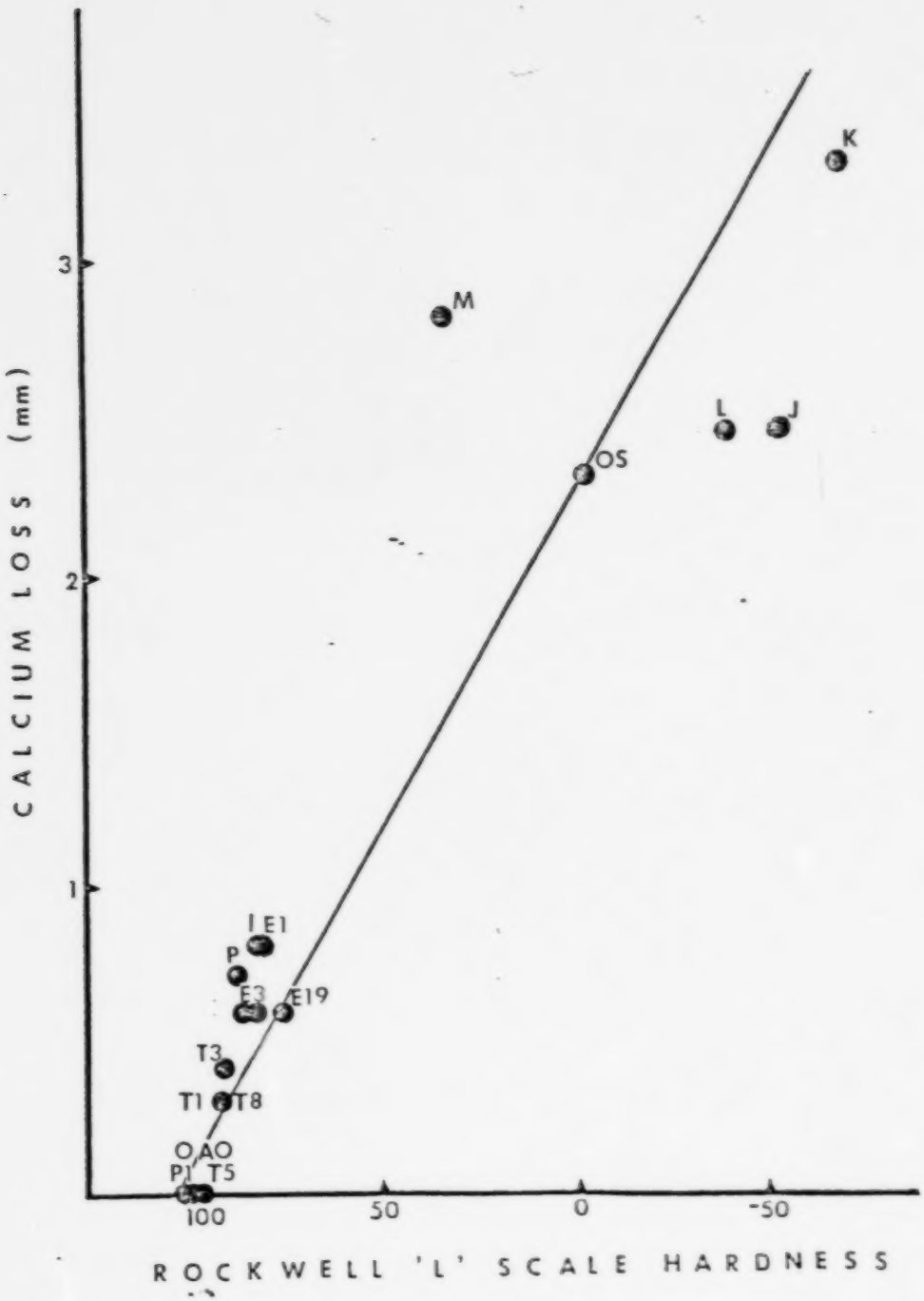


FIGURE 13. Calcium Loss Vs. Rockwell "L" Scale